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Paper and pulp — Deinkability test for printed paper products

Papier et pâte à papier — Essai de désencrabilité des produits en papier imprimés

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The types and sources of paper for recycling are manifold. The most significant grades by volume are packaging products from industry, trade and households, followed by graphic papers from households and to a lesser extent from offices. These papers are blends of a variety of individual products. Typical blends of graphic paper for recycling recovered from households contain many different products printed on papers with a high content of wood-containing pulp fibres and a lesser share of woodfree pulp fibres. Graphic paper for recycling originating from printing and converting operations is typically rather pure and may contain just one type of paper (wood-containing or woodfree). Paper for recycling from printing and converting, as well as special grades, constitute only a minor share of the total volume of paper for recycling. Special grades (e.g. liquid packaging or label stock release liners) sometimes require specific treatments during recycling.

Deinking, the removal of ink from the substrate, is an important step in reprocessing graphic paper for recycling to new paper. A wide variety of papers are produced entirely or partially from deinked pulp and these include:

— graphic papers (of different quality levels);

— hygienic papers (such as toilet paper, hand and kitchen towels);

— white top layers of packaging paper and board.

Good deinkability of printed paper products is crucial for the sustainability of the graphic paper loop. The key process steps for deinking are the detachment of the ink film from the paper, ink fragmentation into a suitable size range and removal from the pulp slurry. Flotation deinking under alkaline conditions is the most widely used technology for ink removal in the paper recycling process. A wider range of the process pH may be utilised for separately collected printed products on predominantly woodfree substrates.

A simplified method herein has been developed to simulate the principle process steps for ink detachment and ink removal under standardised alkaline conditions at a laboratory scale. This gives an indication on how print products will perform in an industrial deinking operation. The method defined in this document is based on INGEDE Method 11. When the first version of INGEDE Method 11 was published, the deinking industry was predominantly using wood-containing raw material. INGEDE Method 11 is widely used by the paper industry and by many stakeholders in the paper value chain. The method is not designed to model additional or alternative process steps, such as dispersing, post-flotation, washing and bleaching. Cleaning and screening stages, which are designed to remove impurities and unwanted materials in the industrial process, are also not included in this method. An alternative deinking test method with near-neutral or neutral flotation conditions may be suitable for paper products mainly consisting of woodfree pulp fibres. However, the near-neutral or neutral flotation conditions are not within the scope of this document.

In most cases, the industrial flotation deinking process is designed and operated to remove a variety of inks and toners. Alkaline pulping conditions and fatty acid based collectors are widely used. However, fatty acid based collector chemistry is not singly used in industrial deinking processes in soft water areas. Assessments based on this laboratory scale method give an indication of how the tested print product will perform in a full-scale alkaline flotation deinking plant, but it will not necessarily provide the same absolute result. An example of this type of relation is given by INGEDE Method 11[3] and the Deinking Scorecard of the European Paper Recycling Council[4].

Paper and pulp — Deinkability test for printed paper products

1 Scope

This document specifies a basic laboratory test method for deinkability, applicable to any kind of printed paper product, under alkaline conditions by means of single stage flotation deinking and fatty acid-based collector chemistry.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 187, *Paper, board and pulps — Standard atmosphere for conditioning and testing and procedure for monitoring the atmosphere and conditioning of samples*

ISO 638, *Paper, board and pulps — Determination of dry matter content — Oven-drying method*

ISO 1762, *Paper, board, pulps and cellulose nanomaterials — Determination of residue (ash content) on ignition at 525 °C*

ISO 2469:2014, *Paper, board and pulps — Measurement of diffuse radiance factor (diffuse reflectance factor)*

ISO 2470-1, *Paper, board and pulps — Measurement of diffuse blue reflectance factor — Part 1: Indoor daylight conditions (ISO brightness)*

ISO 3689, *Paper and board — Determination of bursting strength after immersion in water*

ISO 4119:1995, *Pulps — Determination of stock concentration*

ISO 5269-1, *Pulps — Preparation of laboratory sheets for physical testing — Part 1: Conventional sheet-former method*

ISO 5269-2, *Pulps — Preparation of laboratory sheets for physical testing — Part 2: Rapid-Köthen method*

ISO 5631-1, *Paper and board — Determination of colour by diffuse reflectance — Part 1: Indoor daylight conditions (C/2 degrees)*

ISO 12641-1:2016, *Graphic technology — Prepress digital data exchange — Colour targets for input scanner calibration — Part 1: Colour targets for input scanner calibration*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
deinked pulp
pulp obtained from printed paper products, and deinked according to this document

3.2
un-deinked pulp
pulp obtained from printed paper products, pulped with added deinking chemicals according to this document, prior to flotation

3.3
stock concentration
ratio of the oven-dry organic and inorganic mass of material that can be filtered from a stock sample, to the mass of the unfiltered sample

[SOURCE: ISO 4119:1995, modified — Part of the sentence "when determined as specified in this International Standard" and Note 1 were removed.]

3.4
fibre concentration
ratio of the oven-dry mass of organic material, that can be filtered from a stock sample, to the mass of the unfiltered sample

Note 1 to entry: Organic material is the total material, less its ash.

Note 2 to entry: The organic material mainly consists of cellulosic fibres and fines.

3.5
fibre yield
ratio of the oven-dry mass of organic material after flotation to the oven-dry mass of organic material before flotation

Note 1 to entry: Organic material is the total material, reduced by the oven-dry mass of its ash.

Note 2 to entry: The organic material mainly consists of cellulosic fibres and fines.

3.6
rate of filtration
time taken for a defined volume of a test fluid to pass a filter

4 Principle

Printed papers are subjected to accelerated ageing and then pulping followed by flotation deinking under defined conditions. Pulp samples from each stage are taken and converted to dry state for characterization.

5 Equipment

5.1 General equipment

5.1.1 Drying oven, according to ISO 638.

5.1.2 Analytical balance, up to 150 g with an accuracy of at least 0,001 g.

5.1.3 Balance, up to 3 000 g with an accuracy of at least 0,1 g.

5.1.4 Beakers.

5.1.5 Muffle furnace, according to ISO 1762.

5.2 Equipment for preparation and flotation

5.2.1 Laboratory pulping device, capable of pulping about 150 g to 500 g of paper products under the conditions set in 7.4.1. Examples of suitable devices and operating conditions are listed in Annex A.

5.2.2 Temperature-controlled water bath.

5.2.3 Heating plate, equipped with magnetic stirrer, or commercially available water heater.

5.2.4 Laboratory flotation deinking cell (see 7.6 and Annex B) and – if applicable – accessories.

5.2.5 pH meter, with an accuracy of 0,1 points.

5.3 Equipment for specimen preparation

5.3.1 Pulp distribution device (volume: 10 l).

5.3.2 Büchner funnel.

5.3.3 Vacuum filtration unit for membrane filtration, with 39 mm bottom inner diameter of the funnel.

5.3.4 Vacuum device, that can produce a pressure difference ≥60 kPa.

5.3.5 Filter paper: Grammage of (84 ± 4) g/m^2, filtration time for deionized water (20 ± 4) s, tested according to Annex C and wet burst strength >30 kPa according to ISO 3689.

NOTE 1 The definition of the filter paper is much stricter than in ISO 3688, because the filtrate is used for further analysis (filtrate darkening).

NOTE 2 For example, the filter paper Ahlstrom Munktell 1289[1)] meets these requirements.

5.3.6 Cellulose nitrate membrane filter: nominal ø 50 mm, pore ø 0,45 µm, white, without a grid.

5.3.7 Standard sheet former (model: Rapid-Köthen) with dryer, according to ISO 5269-2 or conventional sheet former according to ISO 5269-1.

5.3.8 Paper cover sheets and carrier boards, according to ISO 5269-2.

5.4 Equipment for analysis

5.4.1 Flatbed scanner or **camera**:

a) Optical scan resolution ≥ 600 dpi, equivalent to a pixel size of ≤42 µm;

b) Colour depth 24 bit;

c) Optical density, D_{MAX} ≥ 4,0;

1) Ahlstrom Munktell 1289 can be obtained at Ahlstrom Germany GmbH, Bärenstein Plant, Niederschlag 1, 09471 Bärenstein, Germany. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

d) with the IT8 calibration (*.ICM-File) according to ISO 12641-1 (see also Annex E IT8 7.2 calibration) and reach a mean grey value of 115 ± 2 for all fields of the IT8 colour calibration sheet according to Annex E.

5.4.2 Image analysis software, such as the ones described in Annex E.

5.4.3 Colour-measuring equipment, which meets the requirements of ISO 2470-1 and ISO 5631-1.

6 Chemicals

6.1 Sodium hydroxide (NaOH), pro analysis, CAS # 1310-73-2.

6.2 Sodium silicate 1,3 g/cm^3 to 1,4 g/cm^3 (38 °Bé to 40 °Bé).

6.3 Hydrogen peroxide (H_2O_2), e.g. 35 %.

6.4 Oleic acid[2] ($C_{18}H_{34}O_2$), purified, CAS # 112-80-1, with the following specifications:

a) acid number: 198 to 240;

b) iodine number: 92 to 100;

c) linoleic acid (C18:2): max. 18 %;

d) oleic acid (C18:1): min. 72 %;

e) palmitic acid (C16:0): max. 8 %;

f) palmitoleic acid (C16:1): max. 1 %;

g) stearic acid (C18:0): max. 4 %.

6.5 Calcium chloride dihydrate ($CaCl_2 \cdot 2\ H_2O$), CAS # 10035-04-8.

6.6 Saturated aluminium sulphate solution $Al_2(SO_4)_3$.

NOTE A concentration of 330 g/l is considered as a saturated aluminium sulphate solution.

7 Procedure

7.1 General

This laboratory scale method defines the essential steps of the flotation deinking process: pulping and flotation. In order to simulate the average age of paper recovered from households, an accelerated ageing step is part of the procedure. Special care was taken to define a procedure without the need to test unprinted paper. The whole laboratory procedure and the required chemicals are shown in Figure 1.

The deinkability is assessed by three main quality parameters of the deinked pulp and one important process parameter.

2) Oleic acid can be obtained at VWR Chemicals, Prolabo, oleic acid, purified, article no. 20447.293. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

Quality parameters:

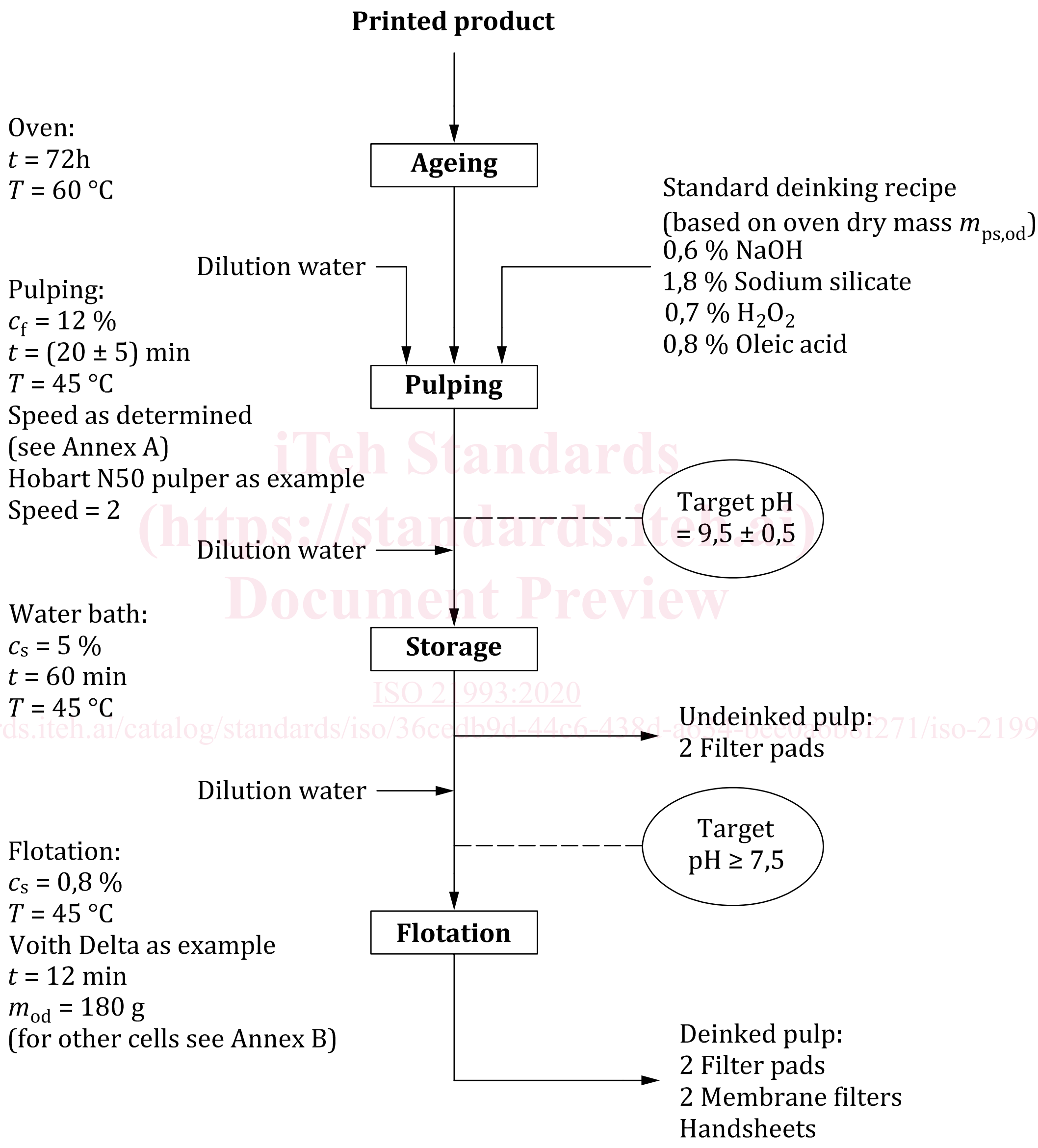
— luminance;

— pulp shade;

— dirt specks.

Process parameter:

— filtrate darkening.


Printed product
Oven:
t = 72h
T = 60 °C
Ageing
Standard deinking recipe
(based on oven dry mass $m_{ps,od}$)
0,6 % NaOH
1,8 % Sodium silicate
0,7 % H_2O_2
0,8 % Oleic acid
Dilution water
Pulping:
c_f = 12 %
t = (20 ± 5) min
T = 45 °C
Speed as determined
(see Annex A)
Hobart N50 pulper as example
Speed = 2
Pulping
Target pH
= 9,5 ± 0,5
Dilution water
Water bath:
c_s = 5 %
t = 60 min
T = 45 °C
Storage
Undeinked pulp:
2 Filter pads
Dilution water
Target
pH ≥ 7,5
Flotation:
c_s = 0,8 %
T = 45 °C
Voith Delta as example
t = 12 min
m_{od} = 180 g
(for other cells see Annex B)
Flotation
Deinked pulp:
2 Filter pads
2 Membrane filters
Handsheets

Key

c_f fibre concentration

c_s stock concentration

t time

T temperature

m_{od} mass (oven dry)

$m_{ps,od}$ mass of the oven-dry printed sample

Figure 1 — Procedure for testing deinkability with standard deinking recipe

